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(54) **Metallurgically bonded diamond-metal composite sintered materials**

(57) A metallurgically bonded diamond-metal composite sintered material suitable for lapping comprises a Ni and/or Co base, an intermetallic compound dispersed in the base and diamond powder. Such a material may be made by sintering nickel and/or cobalt base in powder form having a mesh size of 100 mesh or less together with the element forming the intermetallic compound and the diamond powder at a temperature which is below the temperature at which graphitization of the diamonds occurs. The element forming the intermetallic compound may be selected from one or more of Sn, Sb, Zn, P, S, Mg, Ti, Mo, Se, Ge, In, Te, V, Mb, Ta and B, and the diamond powder content may be 0.1 - 10% by weight.

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FIG. 1

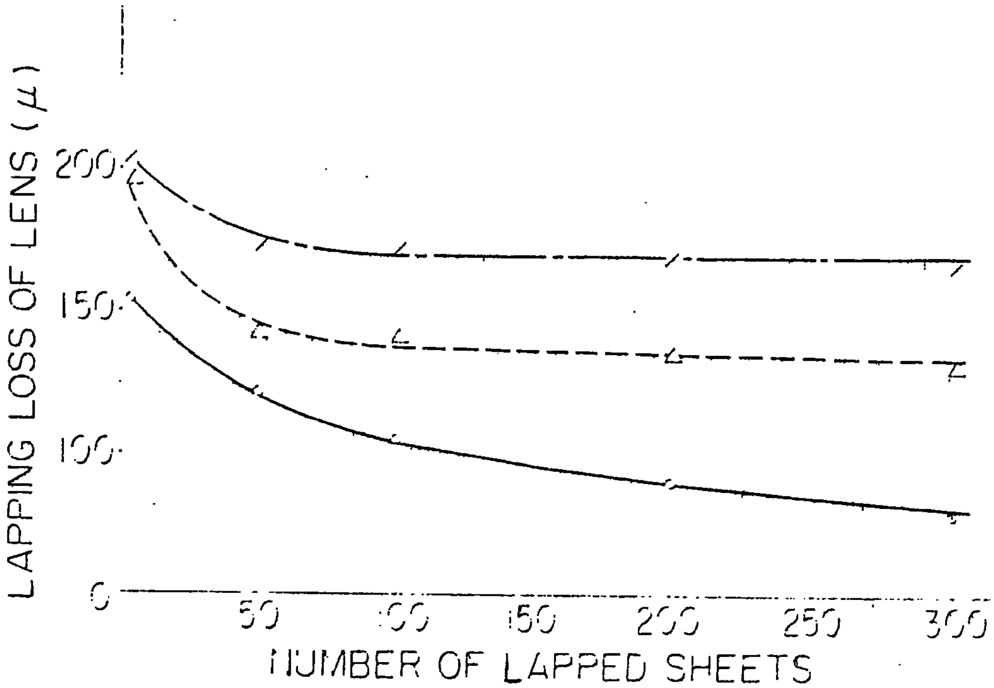

LAPPING LOSS OF LENS (μ)
200
150
100
0
50
100
150
200
250
300
NUMBER OF LAPPED SHEETS

FIG. 2

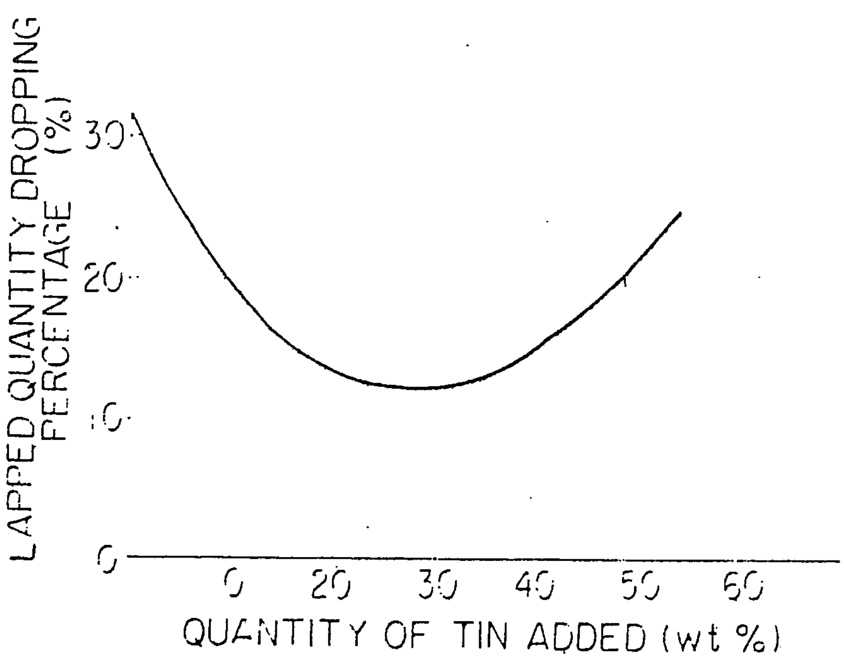

LAPPED QUANTITY DROPPING PERCENTAGE (%)
30
20
10
0
10
20
30
40
50
60
QUANTITY OF TIN ADDED (wt %)

FIG. 3

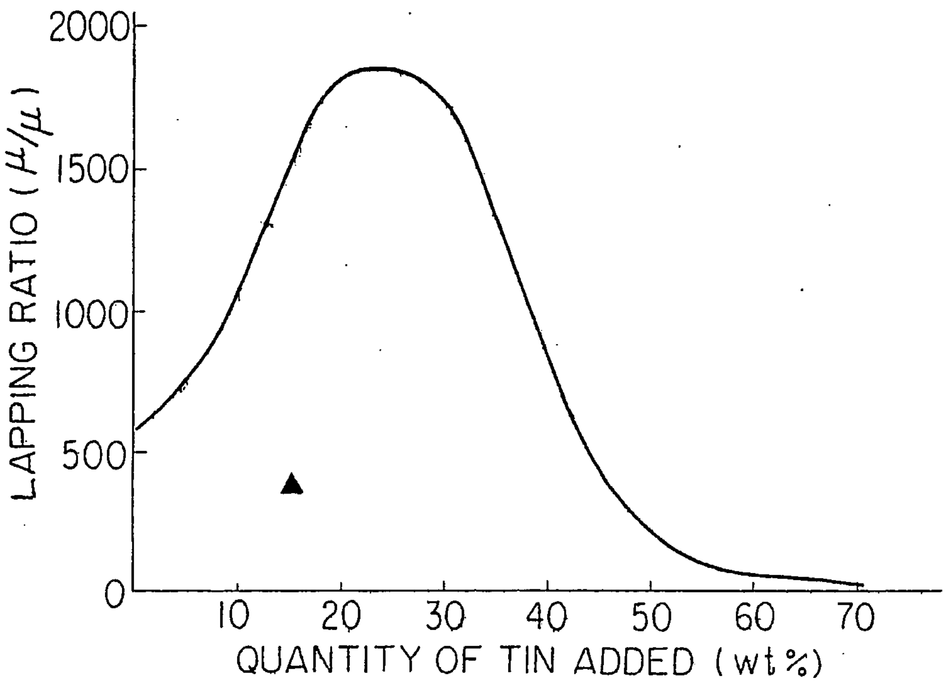

2000
1500
1000
500
0
LAPPING RATIO (μ/μ)
10
20
30
40
50
60
70
QUANTITY OF TIN ADDED (wt %)

FIG. 4

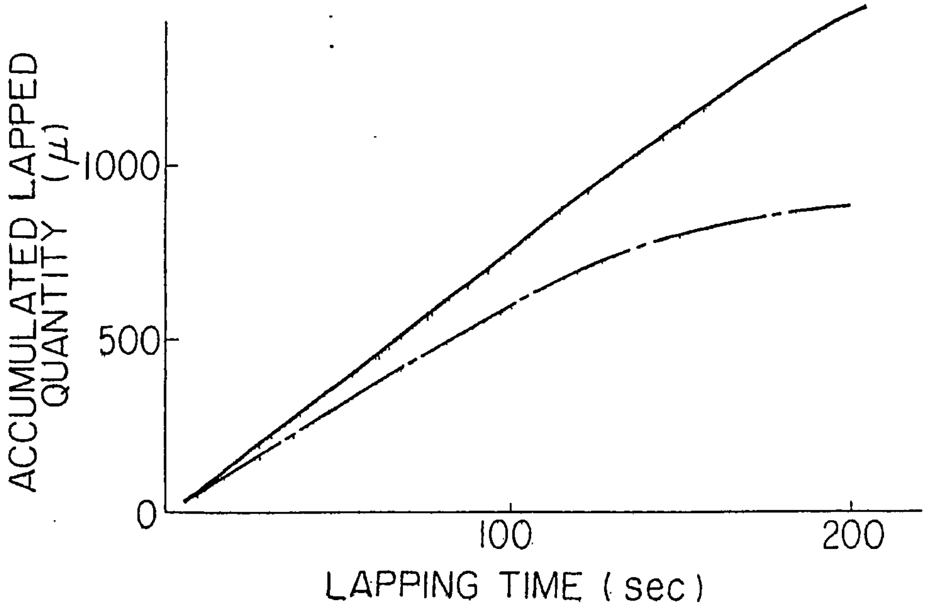
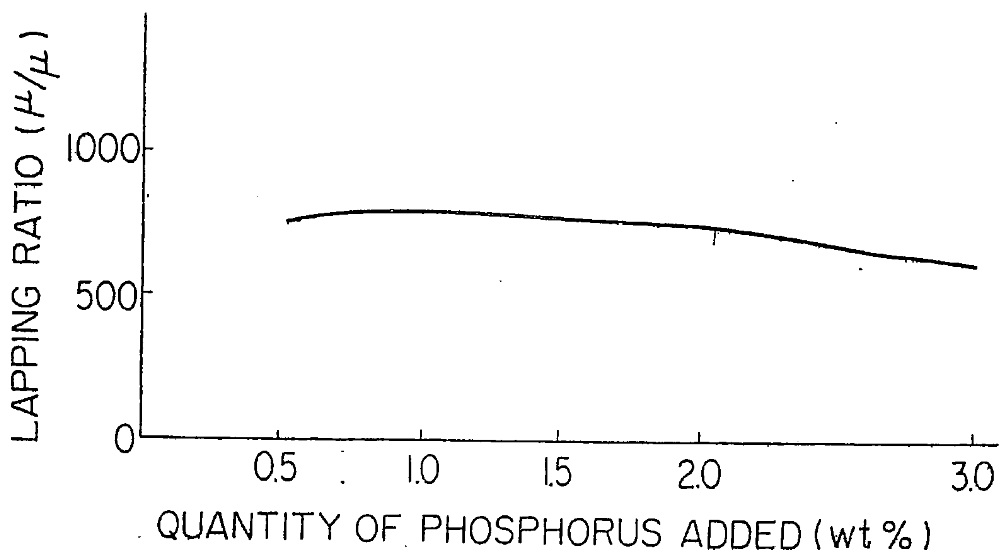

1000
500
0
ACCUMULATED LAPPED QUANTITY (μ)
100
200
LAPPING TIME (sec)

FIG. 5


LAPPING RATIO (H/μ)
1000
500
0
0.5
1.0
1.5
2.0
3.0
QUANTITY OF PHOSPHORUS ADDED (wt %)

SPECIFICATION

Metalurgically bonded diamond-metal composite sintered materials

The present invention relates to metallurgically bonded diamond-metal composite sintered materials, which are suitable for instance for lapping of lenses, and to the production of such sintered materials.

Such sintered materials have recently become widely used for grinding, especially lapping, of spectacle or optical lenses. Copper-tin base alloy diamond-metal composite sintered materials in particular have been used but they have very short lapping lives and they suffer from the risk of a diamond becoming dislodged during lapping, and thus potentially causing serius damage to the surface being lapped. Other base alloy diamond-metal composite sintered materials, such as those where the base alloy is of nickel, cobalt or iron, have been proposed but they suffer from the disadvantage that, due to the high melting point of the alloy, they can only be adequately sintered at temperatures above 1,000°C, and yet such temperatures tend to cause rapid graphitization of the diamonds and consequent damage to the characteristics of the diamonds.

In Japanese Patent Application 159153/1976 we have described how improved sintered materials, having both good diamond-holding force and lapping performance, can be achieved by sintering a composite formed using nickel powder of fine particle size. With this fine particulate nickel powder sintering can be conducted successfully at a low temperature such that the diamond does not graphitize. However the resultant improved products still are not entirely satisfactory for lapping because they become gradually clogged and their lapping properties gradually deteriorate during a long run.

A metallurgically bonded diamond-metal composite sintered material according to the invention comprises a nickel and/or cobalt base, an intermetallic compound dispersed in the base and formed by the addition to the composite before sintering of an element capable of forming the intermetallic compound with the base; and diamond powder.

Such a composite is made by mixing the nickel and/or cobalt powder with the element that will form the metallic compound and the diamond powder, moulding the mix under pressure, and then sintering the mix.

The intermetallic compound dispersed in the base is hard and brittle. The sintered material not only meets the known requirements for lapping or grinding, for instance good diamond-holding force, maintenance of lapping accuracy, prevention of lapping streaks and long life, but also has low tendency to clog during a long lapping run and maintains its lapping performance during a long run. Accordingly the sintered material of the invention is of value in the grinding or lapping of materials such as spectacle and optical lenses, prisms, I.C. boards, watch glasses, marbles and so forth.

The base powder should be sufficiently fine that the sintering temperature can be sufficiently low to avoid the risk of damaging graphitization of the diamond powder. Generally the nickel and/or cobalt powder should be 100 mesh or less in particle size. Suitable nickel powder includes carbonyl nickel, reduced nickel and electrolytic nickel while suitable cobalt powder includes reduced cobalt.

The diamond powder is preferably of 1 micron to 40 micron particle size and is generally used in an amount of 0.1 to 10% by weight of the composite. The diamond powder may be untreated diamond powder or may be diamond powder whose surface has been coated with, for instance, nickel, cobalt, copper or tin. Preferably it is a commercially available electroless nickel-plated diamond powder. The use of such coated diamond powder results in improved strength of the composite.

A variety of elements may be used for forming the intermetallic compond with the nickel and/or cobalt base. Examples include tin, antimony, zinc, phosphorus, sulfur, magnesum, titanium, molybdenum, selenium, germanium, indium, tellurium, vanadium, niobium, tantalum, and boron. The amount of added element must be such that the intermetallic compound is formed during sintering and is dispersed uniformly through the final product and is present in an amount sufficient to give improvement in the lapping properties of the sintered composite. The optimum amount for each added element depends at least in part on the specific gravity of the element being added. Broadly therefore the added elements can be classified into one of two groups. One group consists of the intermetallic compound forming elements of higher specific gravity, such as tin, antimony, zinc, selenium and germanium, and elements of this type are best added in a quantity of 5 to 40% by weight of the total mix. The other group consists of the elements of lower specific gravity, such as phosphorus, sulfur and magnesium and elements of this type are best added in amount of 0.2 to 3% by weight of the total mix. Naturally mixtures of elements from within each group or from different groups may be used.

The preferred method of making the sintered composite comprises mixing 0.1 to 10% by weight of 1 micron to 40 micron diamond powder with the at least one element capable of forming the intermetallic compound with 100 mesh or less nickel and/or cobalt base powder and then moulding and sintering the mix. A small quantity of lubricating agent to facilitate moulding may be included in the mix, for instance zinc stearate or lithium stearate. Moulding is conducted by press moulding in a mould of the desired shape to obtain a compact product, the density of which is preferably in the range 4 to 6.5 g/cc. The compact is then sintered, preferably at 600 to 950°C for 15 minutes to 1 hour, preferably in a non-oxidising atmosphere such as vacuum, hydrogen gas, nitrogen gas or argon gas.

It is readily possible to formulate the mix such that sintering occurs at temperatures below that at which graphitization of the diamond becomes a serious problem. Firstly the base powder can readily be sufficiently fine to permit sintering at temperatures of 600 to 950°C. Further, the formation of the intermetallic compound

formed between the base powder and the additional element occurs during the heating and reduces the sintering temperature needed to achieve a good product.

Tin, antimony, zinc, phosphorus and sulfur have the advantage, as intermetallic compound forming elements, of relatively low melting points. Therefore these elements melt and disperse even at quite low temperatures during sintering and co-operate readily with the base powder to form the intermetallic compounds. After the element has melted dispersed cavities remain in the sintered material and the presence of these cavities enhances the removal of shavings formed during the lapping. The volume of the cavities can be controlled by regulating the quantity of low melting element present in the mix. However higher melting intermetallic compounds can also be used effectively in the invention and also lower the sintering temperature.

It is particularly preferred for the composite to be formed using nickel powder as the base powder and tin as the intermetallic compound forming element.

The intermetallic compound is hard and brittle and thus the sintered material also is hard, and the sintered material has increased abrasion resistance and improved diamond-holding force. During use for grinding or, especially, lapping, clogging is minimised owing to the self-dressing effect of the sintered product during a long run and so the sintered product has improved lapping force retaining properties.

During lapping the diamond powder dispersed in the matrix of base metal and intermetallic compound provides the lapping edges that perform lapping. The contact pressure between the sintered material and the material to be lapped and the number of lapping edges has a close bearing on the lapping efficiency. When a relatively low load or contact pressure is required, e.g. of 30 to 50 g/cm^2, for instance in the lapping of prisms or I.C. boards, the quantity of diamond powder is preferably from 0.1 to 1% by weight so that the number of lapping edges and thus the pressure or load on each edge is appropriate. In particular it is generally preferred that the amount of diamond powder should then be in the range of 0.1 to 0.6% by weight.

The invention is now illustrated by some Examples in which reference is made to the accompanying drawings. In these Figures 1 to 4 illustrate the test results in Example 1, and Figure 5 illustrates those in Example 2;

Figure 1 illustrates the relation between the number of lapped sheets of sintered materials containing tin in varied quantities and the lapping loss of lens wherein the solid line indicates the Ni-0%Sn base sintered material, the dash-dot line indicates the Ni-20%Sn base sintered material, and the broken line indicates the Ni-30%Sn base sintered material;

Figure 2 illustrates the relation between the quantity of tin added and the lapped quantity dropping percentages;

Figure 3 illustrates the relation between the quantity of tin added and the lapping ratio wherein the symbol indicates a Cu-Sn base sintered material;

Figure 4 illustrates the relations in the lapping time and the accumulated lapped quantity between the Ni-25%Sn base sintered material of the present invention and the Cu-Sn base sintered material (Control) wherein the solid line indicates the sintered material of the present invention and the das-dot line indicates the control sintered material;

Figure 5 illustrates the relation between the quantity of phosphorus added to the Ni-diamond-0.1%S base sintered material and the lapping ratio thereof.

Example 1

To a mean particle size 3μ - 4μ carbonyl nickel powder was added 1wt.% of 10 - 20μ artificial diamond powder, followed by the addition of −250 mesh tin in the varied quantities of from 0 to 7-wt.% to obtain mixtures respectively. To these mixtures was further added 0.5 wt.% of zinc stearate. The resulting mixtures were press moulded using a metal mould into $\phi 10 \times H_3$ tablet-like compacts with a compact density of about 6 g/cc. These compacts were sintered in a mixed gas of H_2 and N_2 at 840°C for 45 minutes to obtain sintered materials. The resulting sintered materials were applied for lapping optical lenses with Mohs' hardness of 6 to obtain the results as shown in Figure 1, Figure 2, Figure 3 and Figure 4.

It is noted from these results that in the case tin is not added to the nickel-diamond there is a tendency that the lapping loss of lens decreases as the number of lapped pieces increases, while in the case of the sintered material according to the present invention the lapped quantities do not decrease and moreover the lapping ratio (abrasion loss of less/abrasion loss of sintered material ratio) remarkably increases. As shown in Figure 4, the accumulated lapped quantities decreased with lapse of time in the control sintered material but progressed in a substantially linear manner in the sintered material of the present invention.

Example 2

To a mean particle 3M - 4M carbonyl nickel powder was added 1wt.% of 10μ - 20μ artificial diamond powder, followed by the addition of 0.1wt.% of sulfur and red phosphorus in the varied quantities of from 0.5 to 3wt.%. To these mixtures was further added 0.5wt.% of zinc stearate. The resulting mixtures were moulded according to the same procedure of Example 1 and sintered. The obtained sintered materials were applied for lapping optical lenses with Mohs' hardness of 6 to obtain the lapping ratio. Consequently, the obtained result was as shown in Figure 5.

Example 3

To the mixed powder in which the ratio of a mean particle size −250 mesh or less electrolytic nickel with a mean particle size 3μ - 4μ reduced cobalt powder had been regulated to be 3:7 were added 1wt.% of 15μ - 25μ diamond powder, and further 0.5wt.% of red phosphorus and 0.3wt.% of sulfur. The resulting mixture, upon addition of a predetermined lubricating agent, was moulded according to the same procedure as Example 1 and sintered. The obtained sintered material was applied for lapping the optical lens with Mohs' hardness of 6 to compare the sintered material with the control copper-tin base sintered material in respect of mean lapped quantity and lapping ratio. The obtained results are shown in the following table.

	Mean lapped quantities (μ)	Lapping ratio (μ/μ)
Sintered material according to the present invention	204.1	2354.8
Copper-tin base diamond-metal composite sintered material	139.1	410.6

CLAIMS

1. A metallurgically bonded diamond-metal composite sintered material which comprises a nickel and/or cobalt base, an intermetallic compound dispersed in the base and formed by the addition of the base before sintering of an element capable of forming the intermetallic compound with the base, and diamond powder.
2. A material according to claim 1 in which the intermetallic compound is formed with an element selected from one or more of tin, antimony, zinc, phosphorus, sulfur, magnesium, titanium, molybdenum, selenium, germanium, indium, tellurium, vanadium, niobium, tantalum, and boron.
3. A material according to claim 2 in which the intermetallic compound is formed by the addition of 5 to 40% by weight of one or more of tin, antimony and zinc.
4. A material according to claim 2 in which the intermetallic compound is formed by the addition of phosphorus and/or sulfur in an amount of 0.2 to 3% by weight.
5. A material according to any preceding claim in which the diamond powder is 1 micron to 40 micron in size and is present in an amount of 0.1 to 10% by weight.
6. A material according to claim 5 in which the amount of diamond powder is 0.1 to 1% by weight.
7. A material according to any preceding claim in which the diamond powder is coated with nickel, copper, cobalt or tin.
8. A material according to any preceding claim comprising nickel base and an intermetallic compound dispersed in the nickel base formed by the addition of tin to the base before sintering.
9. A material according to any preceding claim in which the nickel and/or cobalt base had, before sintering, a particle size of 100 mesh or less.
10. A material according to claim 1 substantially as herein described.
11. A method of making a metallurgically bonded diamond-metal composite sintered material which comprises mixing 0.1 to 10% by weight of 1 micron to 40 micron diamond powder with nickel and/or cobalt base powder having a particle size of 100 mesh or less and at least one element capable of forming an intermetallic compound with the base powder during sintering, press moulding the mixture and then sintering the mixture in a non-oxidising atmosphere at a temperature of 600 to 950°C for 15 minutes to 1 hour.

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